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Indian Standard

SPECIFICATION FOR SILVER NITRATE, PURE AND ANALYTICAL REAGENT

(First Revision)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Rs [redacted] Gr 4

February 1978

Indian Standard
SPECIFICATION FOR SILVER NITRATE, PURE AND ANALYTICAL REAGENT
(*First Revision*)

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(*Continued on page* 2)

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Indian Standard

SPECIFICATION FOR SILVER NITRATE, PURE AND ANALYTICAL REAGENT

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 17 October 1977, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1962 under the title 'Specification for silver nitrate, technical and analytical reagent'. Later, with the publication of IS:2318-1974* and imposition of more stringent requirements by other user industries, the Sectional Committee decided to revise the standard and bring it in line with current standards.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for silver nitrate suitable for use in electroplating and other chemical processes (other than photofilms production) and as analytical reagent.

2. GRADES

2.1 There shall be two grades of the material as follows:

a) *Pure Grade* — suitable for electroplating, silvering of mirrors and other processes, other than photofilms, and

b) *Analytical Reagent Grade* — suitable for use in chemical analysis.

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*Specification for silver nitrate, photographic grade (*first revision*).

†Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 Description — The material shall be in the form of colourless crystals free from dirt, foreign matter and visible impurities. The material shall correspond in composition essentially to the formula $AgNO_3$.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR SILVER NITRATE, PURE AND ANALYTICAL REAGENT

(*Clauses* 3.2 *and* B-5.1)

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST REF TO CL NO. IN APPENDIX A
		Pure Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Silver nitrate (as $AgNO_3$), percent by mass, *Min*	99·8	99·9	A-2
ii)	Matter insoluble in water, percent by mass, *Max*	—	0·003	A-3
iii)	Alkalis and other metals (as sulphates), percent by mass, *Max*	—	0·05	A-4
iv)	Bismuth and lead (as Pb), percent by mass, *Max*	—	0·001	A-5
v)	Copper (as Cu), percent by mass, *Max*	—	0·0002	A-6
vi)	Iron (as Fe), percent by mass, *Max*	—	0·0003	A-7
vii)	Chloride (as Cl), percent by mass, *Max*	0·001	0·0005	A-8
viii)	Sulphates (as SO_4), percent by mass, *Max*	0·02	0·0025	A-9
ix)	Matter insoluble in alcohol	—	To pass the test	A-10

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable dark coloured air-tight containers as agreed to between the purchaser and the supplier.

4.2 Marking — The containers, and also the packages where possible, shall be marked legibly and indelibly with the following:

a) Name, grade and net mass of the material;

b) Name of the manufacturer or his recognized trade-mark, if any;

c) Date of manufacture and lot number to enable the material to be traced from records.

4.2.1 The material may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of preparing representative samples of the material and criteria for its conformity with this specification shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR SILVER NITRATE, PURE AND ANALYTICAL REAGENT

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF SILVER NITRATE

A-2.1 Reagent

A-2.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

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*Specification for water for general laboratory use (*second revision*).

A-2.2 Procedure — Weigh accurately about 1 g of the material and dissolve in 150 ml of water, heat to boiling and add slowly with constant stirring 2 ml of dilute hydrochloric acid. Boil until the precipitate coagulates and set aside to cool in dark for at least four hours, preferably overnight. Filter through a tared sintered glass crucible (G No. 4) and wash the precipitate with cold water until free from chloride. Dry the precipitate at 130±2°C to constant mass.

A-2.3 Calculation

$$\text{Silver nitrate (as } AgNO_3 \text{), percent by mass} = \frac{118{\cdot}5 \times M_1}{M_2}$$

where

M_1 = mass in g of the precipitate, and

M_2 = mass in g of the material taken for the test.

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure — Weigh accurately 20 g of the material and dissolve in 100 ml of water. Filter the solution through a tared sintered glass crucible (G No. 4) and wash the residue thoroughly with water. Dry the residue at 105±2°C to constant mass and calculate percentage of residue by mass of the material taken for the test.

Note — Dilute the filtrate and washings to 200 ml in a volumetric flask and reserve it for further tests (*see* **A-4**).

A-4. DETERMINATION OF ALKALIS AND OTHER METALS

A-4.1 Reagents

A-4.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-4.1.2 *Concentrated Sulphuric Acid* — *See* IS:266-1961*.

A-4.2 Procedure — Dilute 100 ml of the solution (*see* **A-3.1**, Note) to 300 ml with water, heat to boiling and add sufficient dilute hydrochloric acid (about 12 ml) to precipitate the silver completely. Set aside overnight, filter, evaporate the filtrate to dryness, moisten the residue with a few drops of dilute hydrochloric acid, dilute with 10 ml of water, boil and filter. Add a few drops of concentrated sulphuric acid to the filtrate, evaporate to dryness, ignite gently and weigh.

Note — Reserve the residue for determination of copper (**A-6**).

A-4.2.1 The material shall be taken to have satisfied the requirement of the test if the mass of residue does not exceed 5 mg.

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*Specification for sulphuric acid (*revised*).

A-5. DETERMINATION OF BISMUTH AND LEAD

A-5.1 Reagents

A-5.1.1 *Ammonium Citrate Solution* — Dissolve 100 g of citric acid in 250 ml of water and make alkaline with strong ammonia solution using phenol red paper as indicator. Finally dilute with water to produce 500 ml.

A-5.1.2 *Potassium Cyanide Solution* — 10 percent (*m*/*v*).

A-5.1.3 *Sodium Metabisulphite Solution* — 1·25 percent (*m*/*v*).

A-5.1.4 *Dithizone Solution* — 0·002 percent (*m*/*v*) in chloroform.

A-5.1.5 *Dilute Perchloric Acid* — approximately 1 N prepared by diluting 82 ml of perchloric acid (r.d. 1·70) in 1 000 ml with water.

A-5.1.6 *Dilute Ammonia Solution* — approximately 6 N, prepared by diluting 280 ml of strong ammonia solution (approximately 18 N) to 1 000 ml with water.

A-5.1.7 *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate in 50 ml of concentrated nitric acid and dilute to 1 000 ml with water in a volumetric flask. Further dilute 10 ml of this solution to 1 000 ml in another volumetric flask just before use. One millilitre of this diluted solution is equivalent to 0·01 mg of lead (as Pb).

A-5.2 Procedure — Dissolve 5·0 g of the material in 30 ml of water in a separating funnel and add 4 ml of ammonium citrate solution, 10 ml of potassium cyanide solution and 5 drops of sodium metabisulphite solution. Extract with 3 successive 5 ml portions of dithizone solution. The final extract should be green; if it is not, continue extracting with 5 ml portions of dithizone solution until the last extract is green. Discard the aqueous solution. Combine the extracts in another separating funnel, add 10 ml of dilute perchloric acid, shake for 1 minute and discard the organic layer. To the aqueous extract add 0·5 ml of ammonium citrate solution, 5 drops of sodium metabisulphite solution, 3 ml of dilute ammonia solution and 5 ml of potassium cyanide solution and adjust the solution *p*H between 9 and 10, if necessary by adding a few drops of dilute ammonia solution. Add dithizone solution in increments of 0·2 ml from a 10 ml micro-burette, shaking vigorously after each addition, until the organic phase is no longer red but has a mauve colour indicating the presence of a slight excess of dithizone. Prepare a blank following the above procedure, but omitting the sample and add to this, finally, the volume of dithizone solution as was added to produce the mixed colour in the test solution. Titrate the blank with standard lead solution, shaking vigorously between each addition of titrant, until the colour of the organic layer matches that of the organic layer of the test solution.

A-5.2.1 The sample shall be taken as having satisfied the requirement of the test if not more than 5 ml of standard lead solution are required.

A-6. DETERMINATION OF COPPER

A-6.1 Reagents

A-6.1.1 *Concentrated Hydrochloric Acid* — approximately 11 N.

A-6.1.2 *Diethylammonium Diethyldithiocarbamate Solution* — 0·1 percent (*m*/*v*) in carbon tetrachloride.

A-6.1.3 *Standard Copper Solution* — Dissolve 3·93 g of cupric sulphate in 200 ml of water, add with stirring 50 ml of concentrated sulphuric acid, cool and dilute with water to 1 000 ml. Further dilute 1 ml of this solution to 100 ml with water. One millilitre of the diluted solution is equivalent to 0·01 mg of copper (as Cu).

A-6.2 Procedure — To the residue from the test for alkalis and other metals (*see* **A-4.2,** Note), add 1 ml of concentrated hydrochloric acid and 1 ml of water, cover with a watch glass and digest on a steam bath for 20 minutes. Cool and dilute with water to 50 ml. Use the solution for test in **A-6.2.1** and **A-7.2.**

A-6.2.1 Transfer 25 ml of the above solution to a separating funnel with 5 ml of diethylammonium diethyldithiocarbamate solution. Carry out a control test by extracting similarly 25 ml of water, 1 ml of concentrated hydrochloric acid, 1 ml of standard copper solution and 5 ml of diethylammonium diethyldithiocarbamate solution.

A-6.2.2 The material shall be taken as having met the requirement of the test if the colour produced in the test is not deeper than that in the control test.

A-7. DETERMINATION OF IRON

A-7.1 Reagents

A-7.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-7.1.2 *Potassium Permanganate Solution* — approximately 0·1 N.

A-7.1.3 *Ammonium Thiocyanate Solution* — approximately 7·5 M.

A-7.1.4 *Mixture of Amyl Alcohol and Amyl Acetate* — in equal volumes.

A-7.1.5 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate $[(NH_4)_2SO_4.FeSO_4.6H_2O]$ in water containing 10 ml of concentrated sulphuric acid and dilute with water to 1 000 ml. Then dilute 10 ml of this solution to 1 000 ml just before use (one millilitre of this diluted solution is equivalent to 0·001 mg of iron as Fe).

A-7.2 Procedure — Transfer 15 ml of the solution prepared in **A-6.2** to a 50 ml Nessler cylinder, add 2 ml of dilute hydrochloric acid, 1 drop of potassium permanganate solution and mix. Add 5 ml of ammonium thiocyanate solution and 10 ml of the mixture of amyl alcohol and amyl acetate, shake vigorously and allow the layers to separate. Carry out a control test in another Nessler cylinder using 9 ml of standard iron solution and 6 ml of water in place of the sample and proceed as above.

A-7.2.1 The material shall be taken as having satisfied the requirement of the test if the red colour produced with the material is not deeper than that produced in the control test.

A-8. DETERMINATION OF CHLORIDE

A-8.1 Reagents

A-8.1.1 *Dilute Ammonia Solution* — approximately 6 N. Prepare by diluting 280 ml of strong ammonia solution (approximate 18 N) to 1 000 ml with water.

A-8.1.2 *Dilute Nitric Acid* — approximately 5 N. Prepare by diluting 320 ml of concentrated nitric acid with water to 1 000 ml.

A-8.1.3 *Silver Nitrate Solution* — 0·2 percent (*m/v*).

A-8.1.4 *Standard Chloride Solution* — Weigh accurately 0·165 0 g of sodium chloride, dissolve in water in a 1 000-ml volumetric flask and make up the volume. Further dilute 10 ml of this solution to 100 ml. One millilitre of this diluted solution is equivalent to 0·01 mg of chloride (as Cl).

A-8.2 Procedure — Dissolve 2 g of material of Analytical Reagent Grade or 1 g of pure grade material in 35 ml of water and add a measured volume of dilute ammonia solution until the precipitate just redissolves (about 6 ml). Electrolyze immediately with a starting current of 1A for one hour, protecting the solution from contamination from the atmosphere. Make the solution slightly acid with dilute nitric acid, add 1 ml of acid in excess and transfer to a 50-ml Nessler cylinder. Rinse the beaker and finally dilute the contents in the Nessler cylinder to 50 ml mark. In another 50-ml Nessler cylinder mix 1 ml of standard chloride solution with the same volume of dilute ammonia solution as used above, neutralize with dilute nitric acid, add 1 ml in excess and dilute to 50-ml mark with water. Finally add 1 ml of silver nitrate solution to each of the two Nessler cylinders.

CAUTION — The ammoniacal solution of silver should be electrolyzed immediately to avoid the formation of dangerously explosive compounds of silver with nitrogen.

A-8.2.1 The material shall be taken to have satisfied the requirement of the test if the opalescence produced with the material is not greater than that produced with standard chloride solution.

A-9. DETERMINATION OF SULPHATE

A-9.0 General — In carrying out this test a preliminary separation of sulphate is considered necessary and hence chromatographic procedure has been prescribed.

A-9.1 Apparatus — The apparatus consists of a glass tube approximately 15 cm long and 1·5 cm in diameter, joined to an outlet approximately 10 cm long and 0·5 cm in diameter through a stop-cock. The outlet enters a filter flask through a bored rubber stopper. The flask is connected to a filter pump.

A-9.2 Reagents

A-9.2.1 *Dilute Perchloric Acid* — approximately 1 N (*see* **A-5.1.5**).

A-9.2.2 *Chromatographic Grade Alumina*

A-9.2.3 *Dilute Hydrochloric Acid* — approximately 1 N and 5 N.

A-9.2.4 *Dilute Ammonia Solution* — approximately 1 N and 0·1 N.

A-9.2.5 *Denatured Spirit* — *See* IS : 324-1959*.

A-9.2.6 *Barium Chloride Solution* — 12 percent (*m/v*).

A-9.2.7 *Standard Sulphate Solution* — prepared by diluting 20·8 ml of 0·1 N sulphuric acid with water 1 000 ml. One millilitre of this solution is equivalent to 0·1 mg of sulphate (as SO_4).

A-9.3 Preparation of Chromatographic Column — Mix 250 g of chromatographic grade alumina with 500 ml of dilute hydrochloric acid and allow to stand for 1 hour with occasional stirring. Wash free from fines by repeated decantation with water until supernatant liquid is almost clear after standing for 1 minute. Filter with suction to remove excess water and dry it in an oven at 110°C. Sift the dried material and reject the portion that passes 75-micron IS Sieve.

A-9.3.1 Plug the column above the tap with glass wool and fill it to a height of 10 cm with a slurry of the prepared alumina. Then wash successively with 50 ml of 1 N dilute ammonia solution and twice with 25 ml of 0·1 N dilute ammonia solution and finally with 50 ml of water.

A-9.4 Procedure — Dissolve 2 g of material of Analytical Reagent Grade and 0·5 g of Pure Grade in 50 ml of dilute perchloric acid. Regenerate the column (*see* **A-9.3.1**) by passing through it 50 ml of dilute perchloric acid and pass the prepared solution of the material through the column at a rate of 10 to 15 ml per minute. Elute with 50 ml of dilute perchloric acid, wash with 30 ml of water. Wash out the receiver, rejecting the contents, and place in it 2·5 ml of 5 N dilute hydrochloric acid. Pass through the

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*Specification for denatured spirit (*revised*).

column 10 ml of 1 N dilute ammonia solution and then 26 ml of 0·1 N dilute ammonia solution, collecting the eluate. To the receiver add 10 ml of denatured spirit, mix, add 1 ml of barium chloride solution, mix immediately and allow to stand for 1 hour. Carry out a control test using 0·5 ml of standard sulphate solution for Analytical Reagent Grade and 1 ml for Pure Grade, with 37 ml of water, 10 ml of denatured spirit, and 1 ml of dilute hydrochloric acid, mixing together, followed by 1 ml of barium chloride solution, mixing and allowing to stand for 1 hour.

A-9.4.1 The material shall be taken as having satisfied the requirement of the test if the turbidity produced with the material is not greater than that produced in the control test.

A-10. TEST FOR MATTER INSOLUBLE IN ALCOHOL

A-10.1 Reagent

A-10.1.1 *Denatured Spirit — See* IS:324-1959*.

A-10.2 Procedure — Dissolve 0·5 g of the material in 0·5 ml of water and add 20 ml of denatured spirit.

A-10.2.1 The material shall be taken as having satisfied the requirement of the test if the solution obtained is clear and colourless.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF SILVER NITRATE, PURE AND ANALYTICAL REAGENT

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions shall be observed.

B-1.1 The sampling instrument and the containers for samples shall be such that the material has no action on them. They shall be clean and dry.

B-1.2 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature. As far as possible, the material shall also be protected from light.

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*Specification for denatured spirit (*revised*).

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the same grade of material drawn from the same batch of manufacture shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

NUMBER OF CONTAINERS IN THE LOT	SAMPLE SIZE
Up to 25	3
26 ,, 50	4
51 ,, 150	5
151 ,, 300	6
301 and above	8

B-2.3 The containers shall be selected at random. In order to ensure randomness of selection, a random number table shall be used. For guidance and use of random number table, IS : 4905-1968* may be referred. In the absence of a random number table, the following procedure may be used:

> 'Starting from any container in the lot, count them as 1, 2, 3,………, etc, up to *r* and so on, where *r* is the integral part of N/n (N being the number of containers in the lot and n the sample size). Every *r*th container thus counted shall be withdrawn so as to constitute the required sample size.'

B-3. INDIVIDUAL AND COMPOSITE SAMPLES

B-3.1 From each of the containers selected according to **B-2.2** and **B-2.3** a representative portion of the material sufficient for the tests shall be withdrawn. These samples, representing each of the selected containers, are termed as individual samples.

B-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken. Such portions shall be thoroughly mixed to give a composite sample.

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*Methods for random sampling.

B-3.3 The material constituting each of the individual samples as well as the composite sample shall be stored separately with full identification particulars.

B-4. NUMBER OF TESTS

B-4.1 Test for silver nitrate content shall be conducted on individual samples.

B-4.2 Tests for the remaining characteristics shall be done on composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — For silver nitrate content, the test results shall be noted and their mean ($\bar{X}$) and the range (R), being the difference between the maximum and minimum of test results, shall be computed. For declaring the conformity of the lot in respect of silver nitrate content, ($\bar{X}-0{\cdot}6\ R$) shall be greater than or equal to the minimum specified in Table 1.

B-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics, the test results of the composite sample shall satisfy the relevant requirements specified in Table 1.

INDIAN STANDARDS
ON
ELECTROPLATING CHEMICALS

IS:

330-1968	Chromium trioxide (*first revision*)
1809-1968	Nickel salts for electroplating (*first revision*)
1880-1977	Zinc oxide and zinc salts for electroplating (*first revision*)
2214-1977	Silver nitrate, technical and analytical reagent (*first revision*)
3026-1968	Tin salts for electroplating (*first revision*)
4846-1968	Sodium potassium tartrate (Rochelle salt)
4847-1968	Copper cyanide for electroplating
5003-1968	Cadmium oxide for electroplating
5761-1970	Gold cyanide and gold potassium cyanide for electroplating
6267-1971	Silver cyanide and silver potassium cyanide for electroplating
6358-1971	Potassium and sodium cyanides for electroplating
6588-1972	Gold chloride (chlorauric acid)
7038-1973	Chloroplatinic acid (platinic chloride)
7067-1973	Fluoboric acid and metal fluoborates for electroplating
8590-1977	Indium sulphate for electroplating